



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES

In re Application of )  
WATZENBERGER et al. )  
Serial No. 09/771,651 )  
Filed: January 30, 2001 )  
For: RECYCLING HYDROXYLAMINE-CONTAINING STRIPPER SOLUTIONS

) Art Unit: 1764

) Examiner: Manoharan

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BRIEF ON APPEAL

Sir:

This Appeal is from the Examiner's Final Rejection of claims 1-9, all of the claims remaining in this application.

REAL PARTY IN INTEREST

The real party in interest is BASF Aktiengesellschaft, Ludwigshafen, Germany, as recorded on January 30, 2001, Reel 011491, Frame 0254.

RELATED APPEALS AND INTERFERENCES

To the best of the undersigned's knowledge, there are no related appeals or interferences within the meaning of 37 CFR 1.192(c).

STATUS OF CLAIMS

Claims 1-9 are pending in this application.

Claim 3 stands objected to as being dependent upon a rejected claim. The Examiner considers that this claim would be allowable if rewritten to be in independent form.

Claims 1, 2 and 4-9 stand finally rejected under 35 USC 103(a) as being unpatentable over Watzenberger et al (US 5,837,107).

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STATUS OF AMENDMENTS

An amendment was submitted on August 26, 2002, but stands not entered having been refused entry by the Examiner in an Advisory Action mailed September 12, 2002.

SUMMARY OF INVENTION

The present invention relates to a process for working up aqueous solutions of hydroxylamine and amines. It has not been possible heretofore to work up such solutions by distillation because hydroxylamine is a thermally labile compound which can decompose in an explosive manner. In solution hydroxylamine is stabilized by amines which are present in dilute form. During distillation water is taken off via the top and the amines remain in the bottom. Being a medium boiler, the hydroxylamine accumulates locally in certain regions of the column and concentrations are reached at which the hydroxylamine tends to decompose - which can take spontaneously and explosively under certain circumstances. Chemical decomposition of the hydroxylamine and subsequent working-up of the amines in an aqueous medium are possible. However, appropriate chemicals must be used for this purpose and give rise to costs. Moreover, the decomposition reaction is slow since the hydroxylamine is stabilized by the amines. It is therefore necessary to accept long residence times and it is for this reason that the plants must have relatively large dimensions. It is an object of the present invention to provide a process for working up aqueous solutions of hydroxylamines and amines which permits easier disposal of the waste or, recycling of the individual components to the production process. The applicants have discovered that this objective is achieved by a novel process for working up aqueous solutions of

hydroxylamines and amines wherein the hydroxylamine is stripped from the solution with steam. An aqueous solution of hydroxylamine which has been substantially freed from amines and in which the hydroxylamine can be decomposed substantially more easily owing to the lack of the stabilizing effect of the amines is obtained. The remaining amine-containing aqueous solution can be worked up by distillation, it being possible to recover the amines in pure form (pages 1-2 of the specification).

### ISSUES

Whether claims 1, 2 and 4-9 are rendered obvious by the disclosure of Watzenberger et al.

The applicants acquiesce in the examiner's stated position with respect to the failure of claims 1 and 2 to particularly point out and distinctly claim the subject matter which the applicants regard as their invention, and to the Examiner's objection to claim 3. To this end, the applicants filed a proposed amendment under 37 CFR 1.116 which was denied entry by the Examiner. The proposed amendment to claim 1 simply corrects an obvious mis-spelling. The proposed amendment to claim 2 simply adds a phrase which particularly points out that the bottom product comprises amines, as requested by the Examiner. The proposed amendment with respect to new claim 10 represents the process already presented in original claim 3 but in independent form, as suggested by the Examiner. Therefore, the applicants respectfully urge that the proposed amendment is in compliance with 37 CFR 1.116, and its entry is again solicited.

### GROUPING OF CLAIMS

The applicants have not argued that the claims herein are separately patentable and do not do so here.

### ARGUMENT

It is the position of the appellants that the examiner erred in rejecting claims 1, 2 and 4-9 under 35 U.S.C. 103 (a) as being unpatentable over Watzenberger et al. (hereinafter, Watzenberger '107). Watzenberger '107 discloses a process for working up aqueous solutions of salts of hydroxylamine with mineral acids. The present invention, on the contrary, relates to working up aqueous solutions of hydroxylamine containing amines (e.g., N-methylpyrrolidone). According to Watzenberger '107, a basic substance is added to an aqueous solution of hydroxylammonium salt of a mineral acid which gives an aqueous solution containing free hydroxylamine and the salt which originates from the base cation and the anion present in the hydroxylammonium salt. The hydroxylammonium salt generally used are the hydroxyl-ammonium salts of mineral acids, for example of sulfuric acid, phosphoric acid or hydrochloric acid. The basic substance generally used are ammonia, sodium hydroxide, potassium hydroxide or calcium hydroxide. The presence of hydroxylammonium salts of mineral acids and the addition of an inorganic basic substance to the aqueous solution of hydroxylamine renders the process disclosed by Watzenberger '107 altogether different from the instantly recited process.

The examiner argues at page 2 of paper no. 7 that:

"However, Watzenberger's process is directed to the same hydroxylamine-containing-material and including e.g., ammonia. As one skilled in the art knows, the argued "amines" are groups of compounds derived from ammonia e.g., by substituting organic radicals for the

hydrogen. "All that is required to show obviousness is that applicant make his invention merely by applying knowledge clearly present in the prior art...Section 103 requires us to presume full knowledge by the inventor of the prior art in the field of his endeavor". Applicants fail to delineate method or process steps not shown nor render obvious by the prior art. The starting material and the product obtained maybe new and unobvious, but, PRODUCT does not impart patentability to the PROCESS of Watzenberger. *In re Durden*, 226 U.S.P.Q. 359."

This line of argument by the examiner does not support his position for several reasons: Firstly, the examiner's reference to the similarity of ammonia and amines, in so far as it is understood by the applicants, does not appear to be relevant to any issue in the present situation. Ammonia is a basic substance which is added by Watzenberger '107 to the aqueous solution of hydroxylammonium salt of a mineral acid to separate out hydroxylamine and the salt which originates from the base cation and the anion present in the hydroxylammonium salt. Even if there is a relationship to be acknowledged between ammonia and some amines, it is not pertinent to the determination of patentability in the instant case. There is no analogy to drawn in the instant case between the use of ammonia by Watzenberger '107 and the presence of amines in the applicants' process.

Secondly, the examiner's reliance on the case of *In re Durden* 226 U.S.P.Q. 359 is misplaced because this is not a situation wherein the applicants are simply applying a known process to a cognate material as in *In re Durden*. The addition of a basic substance to an aqueous solution of hydroxylammonium salt of a mineral acid which gives an aqueous solution containing free hydroxylamine and the salt which originates from the base cation and the anion present in the hydroxylammonium salt is not suggestive of the steam-stripping of a solution of hydroxylamine and amines.

CONCLUSION

In summary, the examiner is believed to have erred for the foregoing reasons, and should not be sustained on this appeal.

Please charge any shortage in fees due in connection with the filing of this paper, including Extension of Time fees to Deposit Account No. 11-0345. Please credit any excess fees to such deposit account.

Respectfully submitted,

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APPENDIX

1. A process for working up solutions of hydroxylamine and amines, wherein the hydroxylamine is stripped from the hydroxylamine-containing solution with steam.
2. A process as claimed in claim 1, wherein the solution is passed into a rectification column and the hydroxylamine is stripped by the countercurrent method with steam, with a top product comprising aqueous hydroxylamine and a bottom product comprising amines being obtained.
3. A process as claimed in claim 2, wherein the top product is condensed and is partly recycled to the rectification column, with reflux ratio of less than 0.5.
4. A process as claimed in claim 2, wherein the rectification column is operated at from 0.1 to 1.0 atmosphere.
5. A process as claimed in claim 2, wherein some of the product is vaporized again by means of an evaporator and the vaporous fractions are recycled to the rectification column.
6. A process as claimed in claim 2, wherein water is added to the liquid phase of the rectification column.
7. A process as claimed in claim 4, wherein the rectification column is operated at from 0.8 to 1.0 atmosphere.
8. A process as claimed in claim 6, wherein the water is passed into the bottom of the rectification column.

9. A process as claimed in claim 1, wherein the solution is from the electronics industry.
10. A process for working up solutions of hydroxylamine and amines, wherein steam is passed countercurrently through the solution in a rectification column operated at from 0.1 to 1.0 atmospheres, and a top product comprising aqueous hydroxylamine and a bottom product comprising amines are obtained, and the top product is condensed and is partly recycled to the rectification column with a reflux ratio of less than 0.5.